

From: ADVANCES IN CRYOGENIC ENGINEERING, Vol. 33  
Edited by R.W. Fast  
(Plenum Publishing Corporation, 1988)

#### A MINIATURE CRYOGENIC HIGH VACUUM VALVE\*

J. D. Siegwarth and R. O. Voth

Chemical Engineering Science Division  
National Bureau of Standards  
Boulder, Colorado

#### ABSTRACT

A small high vacuum valve has been built for use in a liquid hydrogen handling system. The valve stem tip consists of a polycarbonate resin which closes against a stainless steel seat. Other features of the valve include a closing mechanism that is helium gas operated and a bellows stem seal that allows the valve to open and close without changing the internal volume.

#### INTRODUCTION

High vacuum valves operating at 20 K are required in a particular liquid hydrogen handling system. Other requirements dictate that the valves must open and close without changing the system volume. The valve must be remotely actuated. For maximum flexibility in locating the valve in the apparatus, the valve actuator has also been located at cryogenic temperature. To minimize the heat introduced by the actuator, the closing force required to seal the valve should be low. One method of reducing the required closing force is to use a valve with a small port, hence a small surface area of seat contact. This was possible since a high valve conductance was not a requirement for this application. The line sizes to be valved are 1.5 to 3 mm O.D.

Though commercially available valves can meet the high vacuum requirements, they are larger than desired and do not meet the zero volume change requirement. A small, commercially available valve fitted with a driver has been demonstrated to produce a high vacuum seal in cryogenic service.<sup>1</sup> This valve has a 7.9 mm diameter orifice, much larger than is needed for this liquid hydrogen system valve. Besides the fact that it was not a zero volume change valve, the 170 kg load required to close it would be expected to introduce a large amount of heat especially if the driver is at cryogenic temperature. Commercially available valves then, are not suitable for this liquid hydrogen handling system.

---

\*Contribution of the National Bureau of Standards. Not subject to copyright in the U.S.A.

The need for zero volume change when the valve is actuated requires that the stem seal be at liquid hydrogen temperature regardless of the location of the valve actuator. At the low temperature, the stem seal can only be a metal bellows or diaphragm. A lever passing through a bellows or diaphragm seal near its fulcrum is one means of providing the zero volume change desired. Axial motion through a balanced pair of bellows was chosen for the zero volume change seal for the valves described here.

#### VALVE DESIGN AND CONSTRUCTION

Several valve designs were tested before arriving at the design shown in figure 1. This valve features a stem tip of polycarbonate resin (Lexan) seating against a polished stainless steel surface. The stem tip is a 60° included cone. The stainless steel seat is a matching cone. The diameter of the large end of the seat cone is 0.75 mm and the port diameter is 0.4 mm. The stem tip is held to the lower stem assembly by a brass cup that also centers the stem tip. The surface of the valve stem behind the stem tip is curved as shown in figure 1 to permit some self aligning of the stem tip into the seat when the valve is closed. The spring under the stem guide disc provides the force required to open the valve since this lower stem assembly is not connected to the portion of the stem above the bonnet.

The seal between the bonnet and the valve body is a 0.13 mm thick teflon gasket. This is a modification of a stepped seal design reported in the literature.<sup>2</sup> The design shown in figure 1 replaces the stepped seal design with a tongue and groove design. This design prevents the gasket from flowing when the joint is assembled. Rather than machining the groove in a single piece, the outer wall of the groove is provided by the close fitting sleeve shown in figure 1. This design makes the joint easier to machine to a close fit since the inner and outer surfaces of the groove may be separately fit to the inner and outer surfaces of the tongue. The gasket is reusable and easy to remove and replace when so desired. This joint has been disassembled an estimated one hundred times during repeated tests of the valve without replacing the Teflon gasket. Six 8-32 screws clamp the flanges together.

Two identical, welded stainless steel bellows form the stem seal. One connects the bonnet to the lower side of the closing yoke. The other connects the upper side of the closing yoke to the top support, figure 1. The top support is fixed to the bonnet. The internal volumes of the bellows communicate with the internal volume of the valve body. When the closing yoke assembly is pressed down, the upper bellows extends by the same amount the lower bellows compresses. The internal volume of the valve remains unchanged as the valve closes because the volume of a bellows is proportional to the length of the bellows over their operating range. This is true provided the internal pressure does not change enough to bulge the convolutions of the bellows.

An air driven piston at ambient temperature, connected via a stainless steel stand-off tube to the closing yoke, closed the valve during earlier tests of the design. The valve was closed during the final tests by a closely-coupled bellows piston at cryogenic temperature. This cryogenic temperature valve actuator is illustrated in figure 2. It consists of a stainless steel welded bellows, with the top end closed, fitted inside a close fitting brass cup. The lower end of the bellows is soldered to the cup so that the space between the cup and the bellows is a closed volume. The closing yoke fits inside the bellows as shown and the actuator is attached to the valve via the two mounting bolts as shown in figure 2. Introduction of pressurized helium gas into

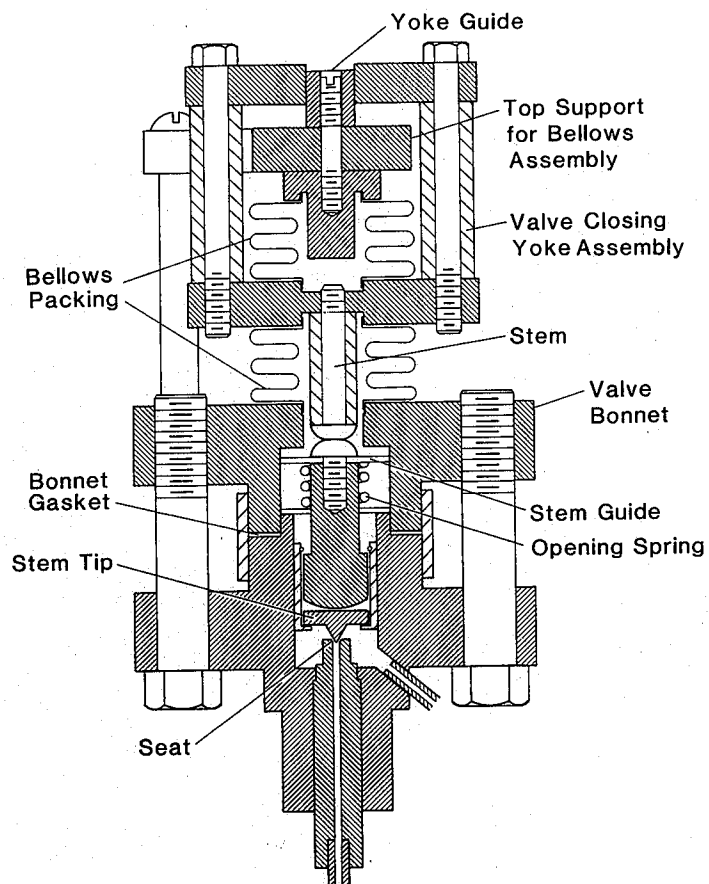


Fig. 1. Cross section of final valve design. The drawing is approximately to scale. The diameter of the bonnet and body flanges are 23 mm.

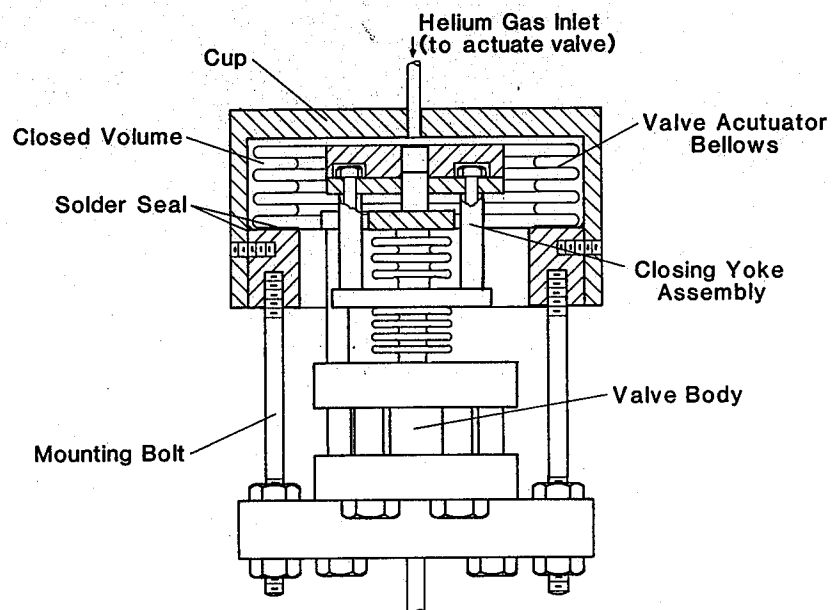


Fig. 2. A cutaway drawing of the cryogenic actuator tested with the valve.

the closed volume compresses the bellows and causes the closed end of the bellows to press down on the closing yoke and force the valve shut.

When the actuating gas is not cooled before it enters the actuator, the heat introduced is proportional to the mass of gas required to close the valve. The added mass of gas is proportional to the pressure required to close the valve and the travel distance of the stem so both should be kept small as possible. The travel was less than 1 mm. The dead volume of the actuator, which is the volume remaining when the bellows contacts the top of the cup, also increases the heat load because that volume must be pressurized. The actuator design of figure 2 minimizes the dead volume.

The force required to seal a valve in low pressure applications depends on the fit between the stem and seat surfaces. Large scale mismatches require seating forces high enough to distort the seating surfaces into conformity. If the large scale fit is perfect, the seating load required should be governed by the surface roughness. A soft stem tip can readily flow to conform to a seat and leak less than  $10^{-8}$  std.  $\text{cm}^3/\text{s}$  of Helium gas at ambient conditions at ambient temperature. At liquid hydrogen temperature materials are much harder and deform less readily. Thus, better sealing surface finishes are required at low temperature than at ambient temperature.

The polishing techniques used for this valve were not at the state of the art. After the seat surface was machined, it was smoothed with 150 grit paper followed by 500 grit paper. Then it was polished with  $1\text{ }\mu\text{m}$   $\text{Al}_2\text{O}_3$  on a swab or on a wooden point on a swab stick. The finished surface appeared polished when viewed under a 30 power microscope.

Chlorotrifluoroethylene (Kel-F) is a commonly used seat material in cryogenic valves. Polycarbonate resin was, however, chosen for this valve because it was easier to machine and polish by the methods used. Like other plastics used in low temperature applications, polycarbonate at 77 K does not shatter when struck. The polycarbonate stem points were machined, then smoothed with 500 grit paper, and polished with jewelers rouge. The final polishing was done with a toothpaste containing an abrasive. This polishing, done in the lathe, left concentric grooves around the tip that were readily visible with a 30 power microscope. A better finish has been obtained using a water soaked cotton swab spun by a 30,000 rpm grinder motor while the stem point was spun in a lathe. The angle between the lathe and grinder axis was held in the 30 to 60° range. This produced a smoother stem tip surface without concentric grooves. However, the surface was still not free from scratches when viewed under the 30 power microscope.

#### TESTING METHODS

In one of the applications, the valve must seal at ambient temperature then remain tight as the valve is cooled to the operating temperature. In another application, the valve must close and remain vacuum tight at cryogenic temperature. Both conditions were simulated in liquid nitrogen tests. A helium leak detector attached to the seat side of the valve was used to measure the leak rate through a closed valve when about  $10^5$  Pa (1 bar) of helium was introduced to the stem side. The estimated sensitivity of the detector was  $3 \times 10^{-8}$  std.  $\text{cm}^3/\text{s}$ . The valve was closed and leak tested at ambient and during the cooling to 77 K in liquid nitrogen. The stem side of the valve was then pumped out and purged with nitrogen gas prior to opening it. The opening was confirmed by the sound of the forepump on the seat side pumping nitrogen gas. The valves were then closed and leak tested again. The valve was

generally opened, closed and leak tested several times while cold. If a valve began to leak, the closing load could be increased by increasing the gas pressure in the valve actuator. The closing load was calculated from the piston area of the valve actuator and the gas pressure. The closing load given for various valves in the next section is the maximum value of the load required during the leak testing to seal the valve. The actual load on the seat could be 20% lower. About 20% of the load was required just to compress bellows and springs.

A different procedure was used for the 20 K tests. During the cool-down, the valve remained open with  $10^5$  Pa (1 bar) of hydrogen filling the system. This pressure was maintained to prevent air from entering and contaminating the valve should leaks be present. When the valve had cooled to 20 K, it was pumped out, closed, filled with  $10^5$  Pa (1 bar) of helium gas on the side opposite the leak detector and leak tested. The valve was then purged with hydrogen to remove the helium, opened, closed and retested. This was done only once because the helium removal was very slow at 20 K, perhaps because of adsorption of the helium.

The zero volume change feature of the stem seal was tested by filling the valve completely with ethyl alcohol with the fill level visible in a vertical 1/2 mm diameter glass capillary connected to the valve. The change in alcohol level as the valve actuated was measured with a cathetometer with a 0.01 mm resolution.

#### VALVE TEST RESULTS

The alcohol level in the measurement of the valve volume change drifted over the period of measurement, which limited the accuracy. The internal volume change as the valve was actuated was less than 0.02 mm, the estimated sensitivity of the measurement.

The final valve design is shown to approximate scale in figure 1. The outer diameter of closure bellows assembly, in figure 2, was 41.3 mm. The overall height of the valve was approximately 50 mm.

The valve was tested for 14 closings at liquid nitrogen temperature. The leak rate was less than the minimum sensitivity of the leak detector,  $3 \times 10^{-9}$  std.  $\text{cm}^3/\text{s}$ , when the closing load was greater than 4 kg. The detector sensitivity was determined with a standard leak. At lower closing loads, the valve would sometimes show a detectable leak. As the closing load was reduced further, a small but fairly repeatable leak was obtained. The ambient temperature closing load required was always less than that required at cryogenic temperature.

After the 77 K tests, the valve was disassembled and the stem tip examined under the 30 power microscope. There was no evidence of distortion of the stem tip by the seat.

The valve was closed twice and helium leak tested at 20 K with a maximum closing load of 3.4 kg. No leak was detected.

The load per square mm of seat projected in the horizontal plane perpendicular to the valve axis was about 13 kg. Assuming a seat width of 0.5 mm for the valve in reference 1, the seat loads were the same. A better finish on the plastic stem tip on the valve reported here might reduce that load.

The pressure differential on the closing bellows required to provide the 4 kg load with a bellows of 7.7 cm active area was 50 kPa (7.5 psi) in liquid nitrogen. A lower closing pressure of only 43 kPa

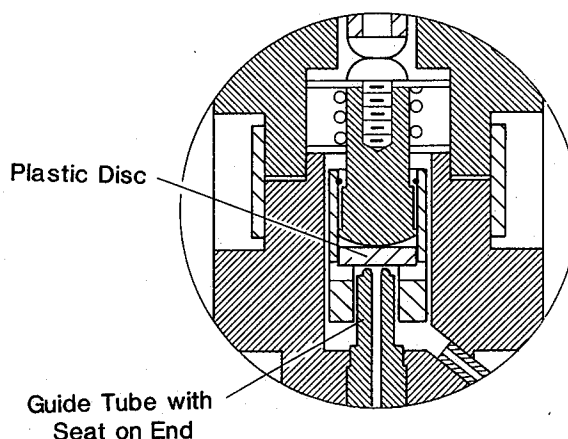


Fig. 3. This shows the alternative seat and stem design tested in the valve shown in figure 1.

(6.2 psi) was sufficient for the 20 K tests. The heat input was detectable when the bellows was activated in liquid hydrogen. Three bubbles of the order of  $1 \text{ cm}^3$  formed. This heat load to the valve actuator could be reduced to essentially nothing by supplying the helium closure gas from a cold reservoir at some less critical location in the apparatus.

#### OTHER DESIGNS TRIED

Some success was obtained with alternate designs, but most were found unsatisfactory. Both coned and flat disc stem tips were tried. Cone shaped stem tips of soft solder, copper, aluminum, filled and unfilled Teflon, and Kel-F were tried. Only the last produced a usable seal. The softness and the poorer machinability of this material, however, caused it to be discarded in favor of polycarbonate resin.

Flat disc stem tips were tested also with some success. Flat discs of some common plastics, Feflon, Kapton and Mylar were tried and failed. In these cases, the stem tip design probably contributed to the failure.

The stem design shown in figure 3 produced some more successful flat disc tests. The extended seat guides and locates the disc to the seat in the design. Some success was achieved with this valve with a chlorotrifluorethylene disc. Some final tests of metal discs were tried with designs shown in figure 3. Indium, 50-50 lead-tin soft solder, unannealed high purity copper and pure gold discs were tried. The first two were soft enough to flow against the seat but would not close leak-tight at 77 K. The copper and gold did not flow significantly. The very slight seat indentation visible was not uniform showing that some misalignment was present in this design. In spite of this misalignment, however, the gold stem disc sealed vacuum tight for eight successive closings at 77 K with a 5 kg closing load. No further effort to determine whether gold might be superior to plastic as a valve stem tip has been carried out.

#### ACKNOWLEDGMENT

This work was supported by Lawrence Livermore National Laboratory.

#### REFERENCES

1. J. F. Siebert, R. A. Hopkins, and H. A. Chameroy, S. H. Castles, "Development of a Launchworthy Motor-Operated Valve for Containment of Superfluid Helium," Proc. 9th Int. Cry. Engr. Conf., Kobe, Japan, 11-14 May, 1982, pp. 182-183.
2. D. N. Astrov and L. B. Belyanskii, "A High Vacuum Seal for Low Temperatures," Instr. Exptl. Tech. USSR, 2, 506 (1966).